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METHODS FOR ESTIMATION OF COMMON PRESERVATIVES USED IN TEXTILE INDUSTRY

PART 3

UDC 677·014·333·2

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 *September* 1983

AMENDMENT NO. 1 SEPTEMBER 1989
TO
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PART 3

(*Cover page, pages* 1 *and* 3, *title*) — Substitute the following for the existing title:

‘ *Indian Standard*

METHODS FOR ESTIMATION OF COMMON
PRESERVATIVES IN TEXTILES

PART 3 ’

[*Clause* **0.3**, (c), (e), (t) and (u)] — Delete.

(*Clause* **1.1**, *line* 3) — Substitute ‘ preservatives in textiles ’ *for* preservatives used in textile industry ’.

Indian Standard

METHODS FOR ESTIMATION OF COMMON PRESERVATIVES USED IN TEXTILE INDUSTRY

PART 3

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Indian Standard

METHODS FOR ESTIMATION OF COMMON PRESERVATIVES USED IN TEXTILE INDUSTRY

PART 3

0. FOREWORD

0.1 This Indian Standard (Part 3) was adopted by the Indian Standards Institution on 29 July 1983, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

0.2 During storage or in use, most of the textile materials are liable to suffer damage as a result of attack by micro-organisms, fungi and bacteria. Numerous treatments have been developed for textile materials with different preservatives (fungicides and insecticides) to protect the material from staining and degradation arising from attack of micro-organisms and insects.

0.2.1 Orthophenyl phenol (OPP) and tributyltin oxide (TBTO) is mainly used for protection of textiles in store and OPP is also used for temporary protection in outside use. Halogenated diphenyl urea derivatives (HDUD) is used for the working life protection of wool and other animal fibres and their blends from moth and beetle damage, during storage and use. Tar oil is used for jute fabrics, sacks, cordages, ropes and nets. Dichlorophen is used for cotton, flax, jute fabrics used as covers, convasses, tarpaulins, awnings, shop blinds, tentage, etc; either alone or incorporated in water and other proofing treatments. It is also used for pressed felts, woollen fabrics, carpet backings, ropes, cordages, slacks, sewing threads, hospital textiles, mattress covers, coated fabrics, mosquito nets, nettings and textiles for aeronautical application.

0.3 Methods for the estimation of the following preservatives have been covered in Parts 1* and 2† of this standard:

a) Salicylanilide
b) Salicylic acid
c) β-Naphthol
d) Pentachlorophenol
e) Creosote
f) Sodium silicofluoride

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*IS : 3522 (Part 1)-1966 Methods for estimation of common preservatives used in textile industry, Part 1.

†IS : 3522 (Part 2)-1970 Methods for estimation of common preservatives used in textile industry, Part 2.

g) Zinc chloride
h) Zinc naphthenate
j) Copper naphthenate
k) Copper-8-quinolinolate
m) Copper salicylanilide
n) Paranitrophenol
p) 2:4 Dinitro-alpha-naphthol (DAN)
q) 4:6 Dinitro-ortho-cresol (DNOC)
r) Dichloro diphenyl trichloroethane (DDT)
s) Dieldrin
t) Lauryl pentachlorophenol (LPCP)
u) Mercapto benzothiazol MBT)

0.4 Most of the preservative agents have toxic properties in some measure and should be handled with care. Some of the chemicals used in the methods of test also present particular hazard. So the tests should only be carried out under the supervision of an experienced analyst.

0.5 Considerable assistance has been derived from BS : 2087: Part 2 : 1981 'Specification for preservative treatments for textiles; methods of test', issued by the British Standards Institution.

1. SCOPE

1.1 This standard (Part 3) prescribes methods for quantitative estimation of single known preservative only and not for mixtures of preservatives used in textile industry. The preservatives covered in this standard are:

a) Orthophenyl phenol (OPP)/2-phenyl phenol
b) Tributyltin oxide (TBTO)
c) Halogenated diphenyl urea derivatives (HDUD)
d) Tar oil
e) Dichlorophen

2. SAMPLING

2.1 Lot — The quantity of textile material of definite type and quality delivered to one buyer against one dispatch note shall constitute a lot.

2.2 Unless otherwise agreed to between the buyer and the seller, the number of bundles or pieces to be selected at random from a lot shall be in accordance with Table 1 or Table 2, respectively.

2.3 From each bundle of yarn or piece of fabric selected as in **2.2**, cut out small portions each weighing about 25 g from at least two different parts and mix them. This shall constitute the test sample. While taking the sample, care shall be taken to exclude a sufficient length of yarn or fabric from both the ends.

TABLE 1 SAMPLE SIZE FOR YARN

(*Clause* 2.2)

LOT SIZE (NUMBER OF BUNDLES IN THE LOT) (1)	SAMPLE SIZE (NUMBER OF BUNDLES TO BE SELECTED) (2)
UP to 150	3
151 " 300	4
301 " 500	5
501 " 1 000	7
1 001 " 3 000	8
3 001 " 10 000	9
10 001 and above	10

TABLE 2 SAMPLE SIZE FOR FABRICS

(*Clause* 2.2)

LOT SIZE (NUMBER OF PIECES IN THE LOT) (1)	SAMPLE SIZE (NUMBER OF PIECES TO BE SELECTED) (2)
Up to 100	2
101 " 150	3
151 " 300	4
301 " 500	5
501 " 1 000	7

3. PREPARATION OF THE TEST SPECIMEN

3.1 Cut the test sample into small pieces. Mix all the pieces thoroughly and draw at least three test specimens from among these pieces each weighing about 5 g.

4. CONDITIONING OF TEST SPECIMENS

4.1 Prior to evaluation, the test specimens shall be conditioned to standard atmosphere at 65 ± 2 percent relative humidity and 27 ± 2°C temperature for 24 hours unless otherwise provided for in the agreement between the buyer and the seller (*see* IS : 6359-1971*).

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*Method for conditioning of textiles.

5. QUALITY OF REAGENTS

5.1 Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water (*see* IS : 1070-1977*) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean the chemicals that do not contain impurities which affect the test results.

6. ESTIMATION OF 2-PHENYLPHENOL (OPP)

6.0 Principle — 2-phenyl phenol is extracted from the textile by steam distillation. An aliquot of the extract is treated with diazotized sulphanilic acid which forms a soluble coloured complex with 2-phenyl phenol. The optical density of the complex is measured on a suitable spectrophotometer at 460 nm.

6.1 Reagents

6.1.1 *Orthophosphoric Acid* — 90 percent (*m/m*).

6.1.2 *Sodium Hydroxide* — 80 g/l solution.

6.1.3 *Sodium Hydroxide* — 10 g/l solution.

6.1.4 *Sodium Nitrite* — (0·05 M solution), prepared by dissolving 0·85 g sodium nitrite in water and making up to 250 ml with water. The solution should be prepared fresh before use.

6.1.5 *Sulphanilic Acid* — 0·5 M solution (approx), prepared by dissolving 1·91 g of sulphanilic acid in 250 ml of water.

6.1.6 *Sulphuric Acid* — (4·5 M), carefully dilute sulphuric acid concentrated 98 percent (*m/m*) to four times.

6.1.7 *Diazotized Sulphanilic Acid Solution* — This solution should be prepared immediately before use by adding 1 volume of sulphuric acid 4·5 M to 5 volumes of sulphanilic acid solution, cooling to 10°C and slowly adding to the solution 5 volumes of the sodium nitrite solution.

6.1.8 *2-Phenylphenol Standard Solution (1 g/l)* — prepared by dissolving 0·100 g of the reagent in 100 ml of the sodium hydroxide (10 g/l) solution and made up to 100 ml in a standard flask with sodium hydroxide. 2-phenylphenol standard reference solution, prepared by diluting 10·0 ml of the standard solution (1 g/l) with sodium hydroxide (10 g/l) solution to 100 ml. 1 ml = 100 μg 2-phenylphenol.

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*Specification for water for general laboratory use (*second revision*).

6.2 Procedure

6.2.1 Weigh 5·0 g of the material, cut into small pieces of not more than 5 mm square and place in a 500-ml round bottomed flask, with a suitable glass ground socket. Add 200 ml of water and 3 ml orthophosphoric acid reagent. Connect the flask for steam distillation and steam distill the contents of the flask, ensuring that a constant volume is maintained by applying gentle heat as necessary. Collect 500 ml of the distillate, taking precautions to prevent the distillate becoming warm at any stage of the distillation.

6.2.2 Make up the volume of distillate to a known volume by addition of water If the volume of distillate is 500 ml, take a suitable aliquot of not greater than 25 ml, dilute with water to 30 ml, add 10 ml of the diazotized sulphanilic acid solution followed by 5 ml of sodium hydroxide (80 g/l) solution and make up to 50 ml with water. Allow to stand for 1 hour, then determine the optical density of the solution on a suitable spectrophotometer at 460 nm using water as a blank. Estimate the 2-phenylphenol content by reference to a calibration graph made from known standards of 2-phenylphenol (*see* **6.2.3.2**).

6.2.3 Transfer by burette 1·0, 2·0, 3·0, 4·0, 5·0, 6·0 and 7·0 ml of 2-phenylphenol standard reference solution to a series of 50-ml standard flasks. To each of the seven flasks and to an empty flask add water to bring the total volume to 30 ml. Then add 10 ml of the diazotized sulphanilic acid reagent followed by 5 ml of the sodium hydroxide (80 g/l)solution and make up to 50 ml with water.

6.2.3.1 Allow to stand for 1 hour and determine the optical density of the solutions at 460 nm using the sample containing no 2-phenylphenol in the reference cell.

6.2.3.2 Draw a graph by plotting optical density against concentration of 2-phenylphenol.

NOTE — The range of the method is 0·01% to 1·4% 2-phenylphenol.

7. ESTIMATION OF TRIBUTYLTIN OXIDE (TBTO)

7.0 Principle — The material is subjected to wet oxidation, the tin in acid solution is solvent extracted. The tin is extracted from the organic solvent and the colour developed with catechol violet is measured by a suitable spectrophotometer at a wavelength of 552 nm.

7.1 Reagents

7.1.1 *Toluene* (low in benzene)

7.1.2 *Ammonia Solution* — 5 M reagent.

7.1.3 *Ascorbic Acid* — 5 percent (50 g/l aqueous reagent solution) — The solution should be prepared freshly.

7.1.4 *Hydrochloric Acid* — 5 M.

7.1.5 *Potassium Iodide* — 5 M (830 g/l) reagent solution prepared by dissolving 83 g potassium iodide in 70 ml water, then diluting to 100 ml with water. The solution should be prepared freshly.

7.1.6 *Sodium Hydroxide* — 5 M.

7.1.7 *Sodium Acetate Trihydrate* — 200 g/l reagent solution. Reagent to be free of fungal growth.

7.1.8 *Sodium Hydroxide* — 0·1 M.

7.1.9 *Sulphuric Acid Concentrated* — 98 percent (*m/m*), 18 M.

7.1.10 *Sulphuric Acid* — 4·5 M.

7.1.11 *Nitric Acid Concentrated*, 70 percent (*m/m*) (16 M).

7.1.12 *Tin (IV) Stock Solution* — 0·20 mg/ml Sn, prepared by dissolving 0·100 g pure granulated tin in 20 ml concentrated sulphuric acid. Heat until fumes appear then, cool before cautiously diluting with 150 ml of water. Cool again before adding 65 ml sulphuric acid concentrated, cool and transfer to a 500-ml volumetric flask and make up to volume with water.

7.1.13 *Tin (IV) Standard Reference Solution* — 10·0 μg/ml Sn, prepared by diluting 5·0 ml of Tin (IV) stock solution to 100 ml with water. This solution should be prepared when required.

7.1.14 *Catechol Violet* — 0·05 g/100 ml in water. This solution should be freshly prepared.

7.2 Procedure

7.2.1 Weigh accurately 1·0 g of the material, transfer to a 200-ml Kjeldahl flask, add 10 ml of sulphuric acid concentrated followed by the addition of nitric acid (16 M) 1-2 ml at a time. Apply gentle heat and digest with the further addition of nitric acid (16 M) until all organic matter is completely destroyed. Evaporate until fuming; cool, add 10 ml of water and boil to remove nitric fumes, evaporate again until fuming. Cool, add 30 ml water.

7.2.2 If the tin content is expected to comply with the light process (or less than 0·05 percent tin) transfer the solution, filtered if necessary, to a 100-ml graduated flask using sulphuric acid (4·5 M) and dilute to the mark with this acid.

7.2.3 If the tin content is expected to comply with the normal process (or greater than 0·05 percent tin) transfer the solution, filtered if necessary to a 250-ml graduated flask using sulphuric (4·5 M) and dilute to the mark with this acid.

7.2.4 Take a suitable aliquot of solution prepared as in **7.2.2** or **7.2.3** of not greater than 25 ml and not containing greater than 40 μg of tin and transfer to a 50-ml separating funnel. Add 2·5 ml potassium iodide (5 M) solution, mix and add 10 ml toluene. If, however, the volume of test solution taken exceeds 25 ml, add 1·0 ml potassium iodide for each 10·0 ml solution in excess of 25 ml. Insert the stopper, shake the funnel vigorously for 2 minutes, allow the layers to separate and discard the aqueous phase. Wash the toluene layer by swirling but without shaking with 5 ml of a solution prepared by mixing 25 ml sulphuric acid (4·5 M) and 2·5 ml potassium iodide solution. Discard the washing (After each of the operations using potassium iodide, wash below the separating funnel tap with water. It is advisable to rinse the funnel exit tubes free of potassium iodide since failure to do so may create unsightly stains following subsequent shaking). Add 5 ml of water to the toluene extract and then sodium hydroxide (5 M) solution dropwise with thorough shaking until the toluene layer is colourless. (This usually requires six to ten drops of the reagent), add a further two drops of sodium hydroxide (5 M) reagent, insert the stopper and shake the funnel for 30 seconds. Allow the layers to separate and run the aqueous layer into a 50 ml beaker, ensuring that all liquid below the tap is transferred to the beaker. Add 3 ml sodium hydroxide (0·1 M solution) to the toluene layer, stopper the funnel then invert and shake for 30 seconds. Allow the layers to separate and add the aqueous layer to the contents of the 50 ml beaker. Retain the toluene layer.

7.2.5 Acidify the aqueous layer in the beaker with 2·5 ml hydrochloric acid and decolorise the liberated iodine by the dropwise addition of ascorbic acid solution. Add 2·0 ml catechol violet solution and mix. Wash the toluene layer retained in **7.2.4** by swirling (but without shaking) with 5 ml sodium acetate solution. Add these washings to the contents of the beaker, mix and adjust the *p*H of the solution to 3·8 ± 0·1 with ammonia solution by means of a *p*H meter. Transfer the solution to a 25-ml standard flask, dilute to the mark with water, and allow to stand for 30 minutes. Measure the optical density of the solution on a suitable spectrophotometer at a wavelength of 552 nm using a 10 mm cell.

7.3 Calculation

7.3.1 Calculate the tin content by reference to a calibration graph prepared from known standards of tin.

7.4 Calibration — Transfer by microburettes 0·5, 1·0, 1·5, 2·0, 2·5, 3·0 and 4·0 ml of tin (IV) standard reference solution to a series of 50-ml beaker. To each of these beakers and also to an empty 50-ml beaker add water to bring the total volume to 7·0 ml. To each, add in the order indicated (which must be strictly adhered to) the following reagents: 1·0 ml sodium hydroxide (5 M) solution mix; 2·5 ml hydrochloric acid solution, mix; 2·0 ml catechol violet solution, mix again; then add 5·0 ml sodium acetate solution. Adjust the *p*H of the solution to 3·8 ± 0·1 with ammonia solution using a *p*H meter. Transfer each solution to separate 25-ml standard flasks, dilute each to the mark with water and allow to stand for 30 minutes. Measure the optical density of each of the solutions in a 10-mm cell at 552 nm with the solution containing no tin in the reference cell. Construct a graph relating the optical density against concentration of tin.

NOTE — The graph should be rectilinear and pass through the origin. Since not all batches of catechol violet behave with equal sensitivity towards tin, recalibration is advised when a different batch of catechol violet is used.

8. ESTIMATION OF HALOGENATED DIPHENYLUREA DERIVATIVE (HDUD)

8.0 General — The method is applicable to the determination of HDUD on wool or on the woollen portion of blends of wool with nylon. It can also be used for blends of other animal fibres with nylon.

8.1 Principle

8.1.1 If no fibre other than wool (or other animal fibre) is present, the textile to be analysed is dissolved in aqueous potassium hydroxide solution and the HDUD is hydrolysed to form 3, 4-dichloroaniline.

8.1.2 If the textile consists of a mixture of wool (or other animal fibre) and nylon, the woollen (or other animal fibre) part of the material to be analysed is dissolved in an aqueous 50 percent potassium hydroxide solution and separated from undissolved nylon fibres. The HDUD is hydrolysed to form 3:4 dichloroaniline.

8.1.3 The amine so obtained is isolated from the alkaline solution by steam distillation and is then diazotized and coupled with benzoyl-H-acid to form a red water-soluble dye. The amount of this dye is determined colorimetrically.

8.1.4 This method is not specific to HDUD. Other compounds, such as certain water-soluble dyes, can also produce a red, water-soluble dye by this method, which can, therefore, interfere with the HDUD determination. However, this has not been found to be a serious drawback in practice and the colorimetric method of estimation has been found to be reliable.

8.2 Apparatus

8.2.1 *Apparatus for the Separation of Wool and Nylon Fibres* — (if required).

8.2.1.1 *Waterbath* — capable of maintaining a 175 mm × 30 mm boiling tube at 71 ± 1°C.

8.2.1.2 *Sintered glass funnel* — porosity grade 1.

8.2.1.3 *Buchner filter flask* — 500 ml capacity.

8.2.2 *Distillation Apparatus*

8.2.2.1 *Round bottom flask* — alkali-resistant, of 1 000 ml capacity.

8.2.2.2 *Distillation splash head* — bulb diameter approximately 70 mm, fitted with a 19/9 ground glass joint on the base and a 24/10 ground glass joint at the end of the distillation tube.

8.2.2.3 *Bulb condenser* — fitted with a 24/10 ground glass socket. Length of the cooling jacket 25 to 30 cm.

8.2.2.4 *One-mark volumetric flasks* — 1 000 ml, 250 ml and 100 ml.

8.2.2.5 *Gas burner or electric heater*

8.2.3 *Waterbath* — capable of being controlled at 10 to 12°C.

8.2.4 *Spectrophotometer* — suitable for the measurement of absorbance (optical density) at a wavelength of 503 nm.

8.3 Reagents

8.3.1 *Potassium Hydroxide Solution* — 500 g/l.

8.3.2 *Potassium Hydroxide Solution* — 2·5 M.

8.3.3 *Hydrochloric Acid Solution* — 1·0 M.

8.3.4 *Sodium Hydrogen Carbonate Solution* — 1·0 M.

8.3.5 *Sodium Nitrite Solution* — 0·1 M.

8.3.6 *Benzoyl-H-Acid* — (that is, 1-benzoylamino-8-hydroxynaphthalene-3, 6-disulphonic acid), 0·01 M solution. The solution should be renewed and stored in an amber coloured bottle.

8.3.7 3,4-*Dichloroaniline* — 0·001 M standard solution. Weigh to the nearest 1 mg, 162 mg of 3,4-dichloroaniline and dissolve in 10 ml of 1 M hydrochloric acid solution, heating, if necessary, on a boiling water bath until completely dissolved. Transfer the solution quantitatively to a 1 000-ml one-mark volumetric flask, dilute to the mark and mix well.

8.3.8 *High Vacuum Grease* or *Soft Paraffin* — for joint lubrication.

8.3.9 *Silicone Antifoam*

8.4 Procedure

8.4.1 *Dissolution of Wool* or *Other Animal Fibres*

8.4.1.1 *Wool and nylon mixtures* — If the textile material to be analyzed is a mixture of wool (or other animal fibre) with nylon, proceed as given in **8.4.1.2** to **8.4.1.5**.

8.4.1.2 Put 0·5 ± 0·001 g of the conditioned material to be analysed into a boiling tube. Place the tube on the water-bath controlled at 71 ± 1°C and add 20 ml of the 500 g/l potassium hydroxide solution at about 20°C. Stir with a glass rod for exactly 5 min; within this period the wool dissolves. Filter immediately through the filter funnel into the Buchner filter flask and drain with suction. Rinse the boiling tube and the nylon residue three times with water, using a total of about 100 ml. To improve the rinsing effect, squeeze the residue with the help of tongs and then drain the filter crucible with suction.

8.4.1.3 Dry the nylon residue in the oven at 105 ± 2°C for 3 h, recondition and determine its mass to an accuracy of 0·001 g.

8.4.1.4 Transfer the filtrate to the distillation flask and rinse the Buchner filter flask with about 30 ml of water.

8.4.1.5 Add a few drops of the silicone antifoam and a few chips of unglazed porcelain to the dissolved wool solution in the distillation flask. Grease the ground glass joints with the high vacuum grease or soft paraffin and assemble the distillation apparatus. Place a 100-ml volumetric flask, containing 6 ml of the hydrochloric acid solution, together with a funnel under the condenser. Complete the estimation of HDUD as given in **8.4.2** to **8.4.5**.

8.4.1.6 *Wool and other animal fibres* — If no fibre other than wool (or other animal fibre) is present, proceed as given in **8.4.1.7**.

8.4.1.7 Place 0·5 ± 0·001 g of the conditioned material to be analysed in a distillation flask. Add 140 ml of the 2·5 M potassium hydroxide solution together with a few chips of unglazed porcelain and a few drops of the silicone antifoam.

Complete the assembly of the apparatus as in **8.4.1.5** and the estimation of HDUD as given in **8.4.2** to **8.4.5**.

8.4.2 *Hydrolysis and Distillation* — Heat the contents of the distillation flask (obtained either in **8.4.1.5** or **8.4.1.7**) to the boiling point and maintain at the boiling point for 15 min. Then distil about 90 ml within 20 to 50 minutes. During distillation, ensure that neither the alkaline solution nor any foam passes through the distillation splash head into the condenser. Cool the distillate in the volumetric flask to 20°C, dilute to the mark and mix well.

8.4.3 *Diazotization* — Measure 50 ml of the contents of the 100-ml volumetric flask into a 250-ml volumetric flask and cool to 10 to 12°C. (The remaining 50 ml of the distillate may be used for a check.) Add 1 ml of the sodium nitrite solution (0·1 M) and allow to stand for 10 min, protected from light, while cooling to 10 to 12°C.

8.4.4 *Coupling* — Add 15 ml of the sodium hydrogen carbonate solution (1·0 M) and, while shaking, add 3 ml of the benzoyl-H-acid solution. After 10 min, dilute the solution with water to 250 ml or to a volume more appropriate to the intensity of the colour of the solution.

8.4.5 *Colorimetric Determination of the HDUD* — Measure the absorbance (optical density) of the solution in the spectrophotometer at a wavelength of 503 nm against water in the reference cell and subtract the absorbance of the reagent blank. Determine the content of the HDUD in the tested sample from a calibration graph.

8.5 Preparation of the Calibration Graph

8.5.1 *Preparation of the Standard Solution* — To a series of six 1 000-ml round bottom flasks, transfer measured quantities of the standard 3,4-dichloroaniline solution shown in the following table:

Standard 3, 4-Dichloroaniline Solution	*Corresponding Percentage of HDUD**
ml	Percent
0†	0
4	0·22
8	0·45
12	0·67
16	0·90
20	1·12

8.5.2 *Colour Development* — Treat the contents of each flask as follows.

8.5.2.1 Dilute with approximately 30 ml of water and cool it to 10-12°C. Add 4 ml of the hydrochloric acid solution and 1·5 ml of sodium nitrite solution, shake well and cool once again to 10-12°C. Allow the solution to stand protected from light for 15 min and then add 15 ml of the sodium hydrogen carbonate solution and 3 ml of the benzoyl-H-acid solution. Shake, allow to stand for 10 min, transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix well.

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*Calculated on 5-chloro-2-(2-sulpho-4-chlorophenoxy)-3′,4′-dichlor, N′ N-diphenyl urea sodium salt, 1 H_2O (molecular weight 562·2).

†Reagent blank.

8.5.3 *Colorimetric Measurement* — After colour development, measure the absorbance of the standard solutions in the spectrophotometer at a wavelength of 503 nm against water in the reference cell.

8.5.4 *Plotting the Calibration Graph* — Deduct the absorbance of the reagent blank from those of the standard solution. Plot a graph having the percentage of HDUD as abscissae against the corresponding values of absorbance as ordinates.

8.6 Expression of Results

8.6.1 *Wool and Other Animal Fibres*

$$\text{HDUD percent} = \text{HDUD}_c \text{ percent}$$

where

HDUD_c percent is the HDUD content as obtained from the calibration graph

HDUD percent is the HDUD content of a conditioned sample of the material

8.6.2 Wool/nylon mixtures

$$\text{HDUD percent} = \frac{\text{HDUD}_c\,\% \times 0{\cdot}5}{0{\cdot}5 - g_n}$$

where

HDUD_c percent is the HDUD content as obtained from the calibration graph

HDUD percent is the HDUD content of the wool portion of a wool/nylon mixture sample

g_n is the conditioned mass of the nylon part of a wool/nylon mixture as obtained in **8.4.1.3.**

9. ESTIMATION OF PHENOL CONTENT OF TEXTILES TREATED WITH TAR OIL

9.0 Principle — The phenols in a dried acetone extract of the material are determined by titration of the extract with a standard solution of potassium hydroxide in propane-2-ol. The test is based on the exothermic conversion of acetone to diacetone alcohol which is catalysed by the free potassium hydroxide present at the end point.

9.1 Apparatus

9.1.1 *Automatic Zero Burette* — 100 ml capacity, 0·05-ml divisions, additional fine jet such as hypodermic needle to deliver 0·3-0·5 ml of titrant per minute with a full burette and tap fully open. The needle is connected to the burette by a short piece of flexible tubing to make a butt joint.

9.1.2 *PTFE Sleeve* to prevent titrant attack on joint of burette.

9.1.3 *Titration Vessel* — 100-ml tall, form beaker.

9.1.4 *Thermometer* — 15 to 45°C.

9.1.5 *Magnetic Stirrer* with polyethylene or PTFE coated follower.

9.1.6 *Soxhlet Extraction Apparatus* 60 ml capacity with 150 ml flask B19 neck.

9.1.7 *Liebig Condenser* B19 socket.

9.1.8 *Claisen Head* — B19 joints.

9.1.9 *Anti-bumping Glass Beads*

9.2 Reagents

9.2.1 *Acetone* (dry and as free from acidity as possible).

9.2.2 *Benzoic Acid*

9.2.3 *Toluene*

9.2.4 *Potassium Hydroxide in Propan-2-ol* — (1 M) standard volumetric solution-prepared by dissolving 56 g of potassium hydroxide in dry propan-2-ol and diluting to 1 000 ml with propan-2-ol. Standardize the solution in the following manner.

9.2.4.1 Weigh accurately about 0·4 g of benzoic acid reagent and dissolve in 30 ml of acetone in the titration vessel. Titrate the solution with the potassium hydroxide solution by the method described under **9.3** until a steep temperature rise is obtained. Read the volume of potassium hydroxide used.

1 ml of 1 M potassium hydroxide solution = 0·122 g benzoic acid.

Let G be the mass of benzoic acid taken and X the quantity in ml of potassium hydroxide solution, then

$$\text{Concentration } (M) = \frac{G}{0{\cdot}122 \times X}$$

9.3 Procedure — Weigh accurately about 3 g of the material (more if the tar oil treatment is light) and place directly in the Soxhlet apparatus. Add 90 ml acetone to the flask, and three glass beads (to prevent bumping) and extract for 2 hours or until the acetone becomes clear. After extraction is complete, allow the apparatus to cool, then remove the flask and connect it to the condenser by means of the Claisen head. Distill off 50 ml acetone, add 30 ml toluene to the residual acetone extract in the flask and re-distil, reducing the volume of the extract to about 15 ml. Transfer the residual extract to the 100-ml beaker, and wash out the flask with small amounts of toluene. Transfer the washings to the extract in the beaker and add 50 ml of acetone. Place the follower and thermometer in the beaker and switch on stirrer. Fill the burette with the standardized potassium hydroxide solution, place the discharge end of the hypodermic needle (which is fitted to the automatic burette) below the surface of the contents of the beaker and start the addition of titrant by opening the burette tap fully, stirring continuously. Observe temperature and immediately a rise is evident, switch off the burette and read the volume of potassium hydroxide used (A). A blank titration experiment should be made using similar quantities of reagents as in the actual analysis to determine the correction for acidity in the reagents (B).

9.4 Calculation

$$P = \frac{(A - B) \times M \times 140 \times 100}{1\,000\, W}$$

where

P = percentage of phenol present
A = ml of potassium hydroxide used
B = ml of potassium hydroxide in blank titration experiment
M = molarity of potassium hydroxide
W = mass of sample taken.

NOTE — 140 is taken as the average molecular weight of the phenols present.

10. ESTIMATION OF DICHLOROPHEN (5,5'-DICHLORO 2, 2'-DIHYDROXYDIPHENYLMETHANE)

10.0 The method is applicable to the determination of dichlorophen in textile materials provided that other phenolic materials are absent.

10.1 Reagents

10.1.1 *4-Aminophenazone* — 10 g/l reagent solution, prepared by dissolving 1 g of reagent in 100 ml of buffer solution, *p*H 9·68.

10.1.2 *Buffer Solution* — *p*H 9·68, prepared by dissolving 13·75 g of disodium tetraborate and 1·125 g sodium hydroxide in water and diluting with water to 1 000 ml.

10.1.3 *Potassium Periodate* — 2·5 g/l reagent solution prepared by dissolving 1·25 g potassium periodate in 500 ml of the buffer solution, *p*H 9·68.

10.1.4 *Dichlorophen* — standard reference solution (20 μg/ml) — prepared by dissolving 0·200 g of dichlorophen, purified melting point 175°C in 10 ml acetone and diluting to 100 ml with the buffer solution.

10 ml of this solution diluted to 1 000 ml with buffer solution is 20 μg/ml.

10.2 Procedure — Extract 1 g of the proofed material (or sufficient to contain 10 mg dichlorophen) by boiling with four successive 25-ml lots of the buffer solution: each extraction should be allowed to continue for 7 minutes before decanting from the material. Combine the extracts, cool, add 100 ml buffer solution and make up to 200 ml with water. Filter a portion and take 4·0 ml and place into a 50-ml volumetric flask. Dilute to about 35 ml with buffer solution and add by pipette 1·5 ml of the 4-aminophenazone reagent. Mix thoroughly then add 10 ml of the periodate solution, dilute the solution with buffer solution to 50 ml and mix well. Control the temperature of the reaction within the range of 17-20°C, protect the solution from light and determine the optical density of the solution at 510 nm on a suitable spectrophotometer after not less than 5 minutes using a reagent blank as zero.

10.3 Calibration — Prepare standard solutions by taking 5, 10 and 15 ml aliquots of the dichlorophen standard reference solution, 20 μg/ml, dilute to about 50 ml with buffer solution and proceed as described in **10.2**. Measure the optical density and plot the data on a graph, optical density against μg of dichlorophen.

10.4 Calculate the dichlorophen content from a previously prepared calibration graph (*see* **10.3**).

INDIAN STANDARDS ON CHEMICAL METHODS OF TEST OF TEXTILE MATERIALS

IS:	
199-1973	Methods for estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials (*second revision*)
200-1969	Method for determination of copper number of cotton textile materials (*first revision*)
244-1969	Method for determination of viscosity (or fluidity) of solutions of cotton and regenerated cellulose in cuprammonium hydroxide (*first revision*)
389-1973	Method for estimation of small quantities of sulphuric acid and hydrochloric acid in cotton materials (*first revision*)
647-1965	Methods for determining the desizing efficiency and the relative efficiency of amylolytic enzymes (*revised*)
1383-1977	Methods for determination of scouring loss in grey and finished cotton textile materials (*first revision*)
3522 (Part 1)-1966	Method for estimation of common preservatives used in textile industry
3522 (Part 2)-1970	Methods for estimation of common preservatives used in textile industry, Part 2
4202-1967	Method for determination of chloride content of textile materials
4203-1967	Method for determination of sulphate content in textile materials
4420-1967	Methods for determination of conductivity of aqueous and organic extracts of textile materials
4655-1968	Method for determination of iron and chromium in textiles
5151-1969	Method for evaluating the relative efficiency of wetting agents for mercerization
5152-1969	Method for estimation of benzene-methyl alcohol-soluble matter in textile materials
5449-1969	Methods for determination of water soluble chromate in textile materials
7044-1973	Method for determination of sodium chlorite content in textile materials
7045-1973	Method for determination of hydrogen peroxide content in textile materials
8476-1977	Method for determination of wool content in woollen textile materials
8477-1977	Method for determination of bitumen content in laminated jute bags

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 $kg.m/s^2$
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemen	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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